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This report covers a period from September 28, 1976, to January 27, 1977 and work was completed as of March 1977.

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FOREWORD

The protection of our estuarine and coastal areas from damage caused by toxic organic pollutants requires that regulations restricting the introduction of these compounds into the environment be formulated on a sound scientific basis. Accurate information describing dose-response relationships for organisms and ecosystems under varying conditions is required. The Environmental Research Laboratory, Gulf Breeze, contributes to this information through research programs aimed at determining:

- the effects of toxic organic pollutants on individual species and communities of organisms;
- the effects of toxic organics on ecosystem processes and components;
- the significance of chemical carcinogens in the estuarine and marine environments.

An integral part of these endeavors is the chemical analysis of the pollutants being studied. Assurance that these analyses are quantitatively and qualitatively valid depends in part on the elimination of interferences from extraneous chemicals. One such source of contamination is addressed by this paper.

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Director
Environmental Research Laboratory

ABSTRACT

Chloroform, trichloroethene,and tetrachloroethene as well as two unidentified compounds were adsorbed from contaminated ground water onto anion-cation exchange resins during their regeneration and cleaning at the distributing plant. Contaminants leached into water passed through the resins after distribution resulted in contamination of deionized water supplies of users. Concentrations of chloroform and trichloroethene, greater in filtered water than concentrations observed in contaminated ground water, indicate the ability of resins to concentrate these compounds. Regeneration and cleaning of contaminated resins by commercial procedures with clean water is ineffective. Contaminated resins, which leached 60 µg/l of chloroform into water, leached 47 µg/l after being returned to the distributor and regenerated with clean water. Increased awareness by users of the limits of ion exchange resins together with stricter water quality control of distributors is indicated.

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SECTION 1

INTRODUCTION

Medical, biological, and analytical laboratories routinely use water for the preparation of reagents, dilutions, bioassays, and analytical procedures as well as for rinsing of glassware. Water free from undesirable contaminants is therefore an absolute necessity; thus, much effort is expended assuring that an abundant supply of pure water is available.

In many cases, however, this effort begins and ends with the installation of ion exchange resin filters. Where resins have undergone strict quality control, such as imposed by the manufacturer, the use of demineralizers is sufficient to meet many general laboratory needs. Large units of ion exchange resins that require maintenance or regeneration by distributors, however, are no longer subject to this control and have the potential of becoming a source of contamination. Further, the problems associated with the mistaken but common assumption that deionized water is a substitute for distilled or extracted water become compounded where water free of organics is needed. Ion exchange resins as a source of contamination is exemplified by the following case study.

Ion exchange resin and carbon filters distributed to industry, research laboratories, and private homes for water softening and conditioning purposes were contaminating the water they serviced with volatile chlorinated organics. Ground water contaminated by an unknown source with chloroform, trichloroethene, tetrachloroethene, and two yet unidentified compounds was used by the water conditioning company for the processes involved in cleaning and regenerating the filters, resulting in their contamination prior to distribution to customers.

Efforts to determine the extent as well as the source of the ground-water contamination were undertaken by the Department of Health and Environmental Control of South Carolina.

SECTION 2

CONCLUSIONS

Adsorption of contaminants from polluted waters by ion exchange resins dictates that their quality or freedom from contamination is only as good as the water used to regenerate and clean them.

Many reports recognize our increasing dependence on ground-water resources. They also note the growing evidence which indicates that the nation's ground waters are becoming excessively polluted by various surface and subsurface disposal techniques (Robertson et al, 1974; Van der Leeden et al, 1975; Crouch et al, 1976; Dunlap et al, 1976; Miller et al, 1977; Tinlin, 1976; Todd et al, 1976).

The affinity of ion exchange resins for neutral halogenated organics indicates that once contaminated, standard commercial procedures employed for the regeneration of exhausted resins are inadequate to insure an uncontaminated product. The need for an increased awareness among laboratories of the limits of ion exchange resins as well as their potential for contamination is apparent.

Finally, the sources of potential ground-water contamination are addressed by existing federal and state pollution control programs, but these controls do not provide comprehensive ground-water protection due to the absence of effective monitoring and reporting programs (Easton, 1977). Although such programs would not eliminate the need for improved water quality control among distributors and users of demineralizers, they would help prevent repetition of occurrences similar to the one reported here.

SECTION 3

EXPERIMENTAL

Water samples were collected from the various systems into 100-ml volumetric flasks equipped with teflon-lined screw caps (Table 1). The flasks were brought to volume and immediately extracted by employing a method similar to one reported by Henderson et al (1976). A 5-ml aliquot of pesticide grade pentane (Fisher) was pipetted directly into the flask. The water/pentane mixture was vigorously shaken for approximately five minutes; a 3.0-µl aliquot of the organic layer was then removed with a microliter syringe for chromatographic analysis.

Gas chromatograms were obtained with a Hewlett Packard model 5710A equipped with a ^{63}Ni electron capture detector. A 1.8-meter by 2-mm i.d. glass column, packed with 12% OV-101 on Anakrom Q 100/120 mesh (Analabs), was utilized. Analyses were run at an injection port and detector temperature of 100°C. The column was maintained at 55°C with 95:5 argon/methane mixture as the carrier gas (40 ml/min.).

Chromatographic results of Bears Bluff ground water and Charleston city water extractions were negative. Water standards were prepared from standard solutions of the three compounds of interest in ethanol. The average recovery rate of chloroform was 89%, whereas those of tri- and tetrachloroethene were 91% and 97%, respectively.

Trichloroethene and tetachloroethene assignments for two of five gas chromatographic peaks observed (Fig. 1) were obtained at the EPA's Region IV, Surveillance and Analysis Division laboratory, Athens, Ga., where samples were analyzed by the Bellar Volatile Organic Analysis Technique (Bellar and Lichtenberg, 1974), and quantified on a Tracor 210 gas chromatograph. Mass spectral confirmation was made with a Finnigan 1015.

Chloroform could not be confirmed by mass spectrometry, but good GC evidence was obtained for its identification by employing the admixture technique to an extracted water sample. The resulting chromatograms indicated no new peaks nor shoulder or irregularities on the chloroform peak. An increase in peak-height without corresponding increase in peak-width, conveniently measured at half-height, indicated excellent retention coincidence between chloroform and the unknown. The presence of chloroform was further confirmed by GC analysis at the Department of Health and Environmental Control laboratory in Columbia, S. C. Quantification was accomplished by direct comparison of sample peak areas to those of standards of each of the three identified contaminants.

TABLE 1. DISTRIBUTION AND CONCENTRATION OF CHLOROFORM, TRICHLOROETHENE, AND TETRACHLOROETHENE CONTAMINATION

Sample	Location	Date	$CHCl_3$	C_2HCl_3	C_2Cl_4
			µg/l		
Mt. Pleasant water system Water Conditioning Service tap	Mt. Pleasant, S.C.	11-5-76	<0.1	<0.1	0.7
Ground water (9 meter well) Water Conditioning Service plant	Mt. Pleasant, S.C.	11-5-76	1.6	2.8	383
Process water Water Conditioning Service plant	Mt. Pleasant, S.C.	11-5-76	6.0	12	554
Filtered water, Med. Univ. of S.C. Infectious Diseases Laboratory	Charleston, S.C.	10-6-76	27	<0.1	1.9
Filtered water, Med. Univ. of S.C. Basic Sciences Building, Rm. 527	Charleston, S.C.	10-6-76	60	5.9	5.8
Filtered water,[a] Environmental Analytics Inc.	Goose Creek, S.C.	10-6-76	ND[b]	ND	3.2
Filtered water, EPA Bears Bluff Field Laboratory	Wadmalaw, Is., S.C.	10-6-76	29	54	6.1

[a] Filters in use 6 months

[b] Not detected

Figure 1. Typical Gas Chromatogram of Pentane Extract from Water Passed through Contaminated Ion Exchange/Carbon Filters.

SECTION 4

RESULTS AND DISCUSSION

Having determined that our laboratory ground water was free from contamination, initial experiments indicated that contaminants appeared after the water was passed through a commercial water treatment system designed to furnish deionized water rated at 18 megohm-cm. The system was comprised of one activated carbon, followed by two ion exchange filters which contained a cation-anion resin mixture.

Follow-up tests on water samples from similar water treatment and conditioning systems serviced by the same company showed the same contaminants in systems located at diverse and distant points in Charleston County, S. C. (Table 1). Sampling conducted at the servicing plant revealed the process water used for back flushing, regeneration, and rinsing of the ionic resins and activated carbon employed in the filters was obtained from a 9-meter well contaminated with the same volatile halocarbons. Three of the five observed contaminants (Fig. 1) were subsequently identified as chloroform, trichloroethene, and tetrachloroethene.

Although the types of contaminants remained constant over four months of sampling, there was degree of variability but no apparent trends of their concentrations in the three systems analyzed during this period (Table 2).

TABLE 2. VARIATION IN CONTAMINANT CONCENTRATION OVER SAMPLING PERIOD 9-29-76 TO 1-2-77

Sample	$CHCl_3$	C_2HCl_3	C_2Cl_4
		µg/l	
Ground water service plant	< 0.0-2.8	0.6-2.8	88-383
Process water service plant	1.5-6	< 0.1-7.8	389-594
Filtered water, EPA Bears Bluff Field Lab	< 0.1-38	< 0.1-83	3.1-61

Noteworthy was an apparent increase in concentration of chloroform and trichloroethene within the filters during servicing. While the concentration of these two compounds in the contaminated ground water never exceeded 3 µg/l their concentration markedly increased in filtered ground water. Maximums of 27 µg/l and 60 µg/l chloroform were detected in samples obtained from the Infectious Diseases Laboratory and the Basic Sciences Building filters (Table 1), respectively, and a high of 38 µg/l was detected in our laboratory filters (Table 2).

In general, the length of time various water treatment systems were in use at their respective locations after being serviced had little effect on the concentration of contaminants leached into the water supply. For example, filters which had exhausted their ion exchange capacity after several months of use continued to contaminate our water at approximately 80% of their original rate with 18 µg/l chloroform, 0.1 µg/l trichloroethene, and 50 µg/l tetrachloroethene.

The persistence of these chemicals was demonstrated when filters containing old, but freshly washed and regenerated resin and carbon, leached 47 µg/l chloroform into water passed through this system.

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